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Chi, Lin; Jensen, Ole Mejlhede

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Department of Civil Engineering

Technical University of Denmark

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Lin Chi
Ole Mejlhede Jensen
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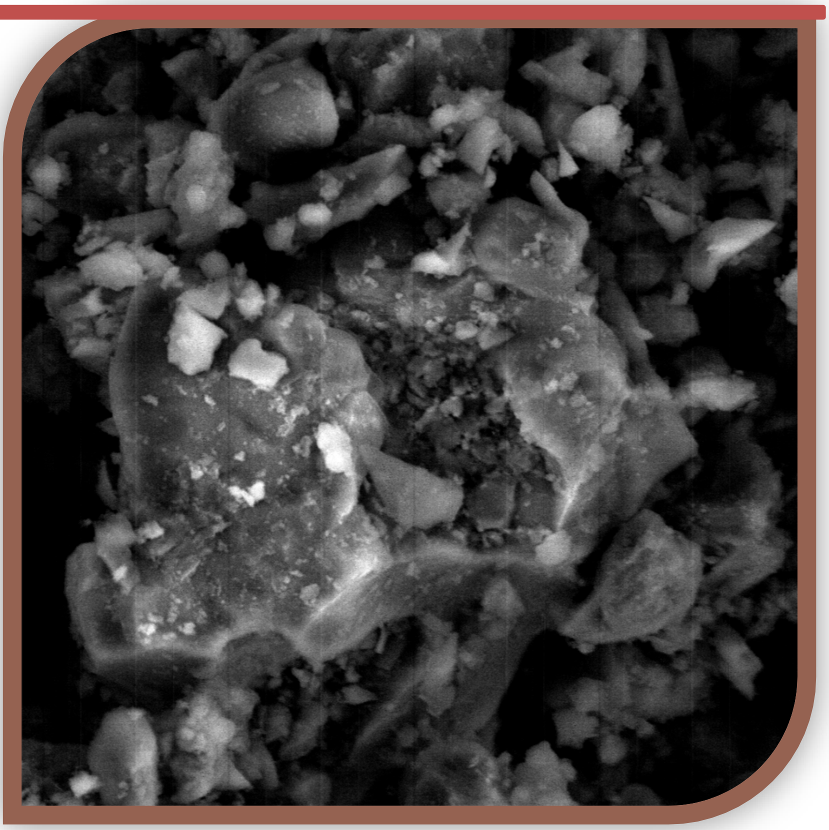


Abstract

The aim of this project is to study the effect of alusilica (ALS) as a mineral admixture on the fresh properties and development of mechanical properties of cementitious systems. ALS consists of relatively pure, amorphous silicium-dioxide – a chemical compound which is known to be useful as mineral admixture in concrete. The project has been carried out in cooperation with the company alufluor (Helsingborg, Sweden), and MSc Ebbe Skyum Jøns.

The application of ALS was investigated as partial cement substitution in mortar. A total of three mortar mixtures were produced: 1) reference, i.e. no substitution of cement, 2) cement clinker and gypsum substituted partly with ALS before grinding (referred to as “co-ground”), and 3) cement (i.e. ground cement clinker and gypsum) partly substituted with ALS and subsequently blended (referred to as “blended”). The level of substitution was 10% ALS relative to the total binder mass (cement+ALS). The water/binder-ratio (w/b) is 0.5 for all mixtures. The produced ALS-substituted powder was studied by scanning electron microscopy (SEM) and Energy Dispersive X-ray Analysis (EDAX) to investigate if the ALS agglomerates in the raw material were broken by the grinding procedure.

On the fresh mortar air content was measured by the pressure method, ASTM C231/C231M-14 and the flow was measured by ASTM C1437-13. Casting was done in standard mortar molds 4×4×16 cm^3. After demolding, each mortar specimen was weighed over and under water to evaluate their homogeneity and air content, and subsequently they were immersed in lime water until further testing. Mechanical testing generally followed EU standard EN 196-1. In accordance with the EU standard, measurements of mechanical properties were done at a minimum of 3 samples at each test time 1, 2, 3, 7, 14, 28, 56 and 112 days. On hardened samples air content was additionally measured by point counting.

The inclusion of ALS in the mortar as a mineral admixture with the cement substitution ratio of 10% resulted in a higher air content and lower flowability in comparison with the reference mortar. Compared with blending ALS during mixing, mortar containing co-ground ALS has properties closer to the reference mortar.

ALS substitution seems to only have a minor effect on the flexural strength throughout the hardening. Mortar with ALS substitution, exhibited a lower compressive strength as compared to the reference mortar. However, a major part of this strength reduction seems to be caused by the lower flowability and the related higher air content. The ALS substituted systems may potentially be optimized through adjustment of a plasticizing agent, and in that case there is not expected to be a strength reduction. It is concluded that ALS can be a useful cement substitution.

For further tests it might be relevant to investigate the performance of ALS-systems at a low w/b, e.g. 0.3 and at higher temperatures, e.g. 40-60°C which is realistic to be encountered also in practice. For such conditions a more clear advantage of ALS as a cement substitution may be present. Additionally it would be relevant to investigate the durability properties of ALS substituted systems.

Contents

1. Background

Alusilica (ALS) is an industrial by-product from the production of aluminium fluoride (AlF_3). ALS consists of mainly amorphous silicon dioxide, but it contains some fluorides (~5 wt%), notably in the form of aluminium fluoride, which may cause problems in cementitious systems. However, this fluoride can be made chemically inert through a simple reaction with CaO.

ALS can potentially be used as a mineral admixture in concrete. The aim of this project is to investigate this possibility. A preliminary investigation took place as a BSc project in 2015 with encouraging results but also with a number of issues which needed further investigation [1]. To study the effect of ALS as a mineral admixture on the fresh properties and development of mechanical properties of cementitious systems, investigations are carried out as follows:

A) Production of three binders: 1) reference (co-ground clinker and gypsum, no substitution with ALS), 2) cement clinker and gypsum partly substituted with ALS before grinding (referred to as "co-ground") and 3) cement (i.e. ground cement clinker and gypsum) partly substituted with ALS and subsequently blended (referred to as "blended").

B) Examination of powders by sieving, air permeability test, scanning electron microscopy (SEM) and Energy Dispersive X-ray Analysis (EDAX).

C) Measurement of fresh mortar properties: air content and workability (flow).

D) Measurement of hardened mortar density (including air content).

E) Measurement of air content by point counting on the hardened mortar.

F) Measurement of mechanical properties, i.e. flexural and compressive strength.

It is hypothesized that co-grinding as opposed to blending the ALS will break ALS agglomerates and thereby improve its interaction in the cementitious systems.

2. Materials

2.1 cement clinker

Aalborg Portland pure cement clinker and gypsum are used. The amounts of the materials delivered are approximately 32 kg of cement clinker and 1.35 kg of gypsum ($CaSO_4 \cdot 2H_2O$). The mineral composition of cement clinker is listed in Table 1.

Table 1. Mineral composition of cement clinker. This clinker is normally used for production of white Portland cement. Note that cement chemistry nomenclature is used for the components.("C"=CaO, "S"=SiO_2, "A"=Al_2O_3, "Fe"=Fe_2O_3).

Component	C_3S	C_2S	C_3A	C_4AF
Content (wt%)	77	16	5	1

2.2 Alusilica

ALS is provided by the company Alufluor, Sweden. The product data sheet for the original material is shown in appendix I. However, the material has been further processed in order to passivate the fluorides which are present mainly as aluminum fluoride: 4.2 kg ALS and 500 g CaO (a large surplus for the reaction) were mixed in hot water. This makes the fluoride react and precipitate as calcium fluoride, CaF_2, which is stable in the cementitious systems. An analysis of the reacted mixture shows that there is only a negligible amount of free fluoride left (<0.5 g). The composition of unreacted and reacted ALS are measured by X-ray fluorescence (XRF) and the result is shown in Table 2.

Table 2. Composition of unreacted and reacted ALS according to XRF. The apparent increase in fluoride content by the stabilization process is most likely due to uncertainty in the analysis.

Content (%)	F	Al	SiO_2	Ca	LOI
Raw ALS	4.9	2.1	85	-	8
Fluoride stabilized ALS	6.2	1.9	73	8.7	10.2

The moisture content in the fluoride stabilized ALS received from the producer is 4.8%. Before use the ALS is dried to a constant mass at 105^{o}C.

2.3 CEN standard Sand

CEN standard sand (EN 196-1, Normensand, Beckum, Germany) is used in bags with a content of (1350$\pm$5) g. The particle size distribution of the sand is given in Table 3.

Table 3. Particle size distribution of the CEN Reference sand according to the producer.

Square mesh sieve size (mm)	2.00	1.60	1.00	0.50	0.16	0.08
Cumulative sieve residue (wt%)	0	7 ± 5	33 ± 5	67 ± 5	78 ± 5	99 ± 1

2.4 Demineralized water

The water was demineralized.

3. Method

3.1 Preparation of mortar

3.1.1 Composition of mortar

Mix proportions are based on Standard DS/EN 196-1 and given in Table 4. The amount of gypsum (5% of cement) is on the safe side to avoid flash set. The water/binder-ratio is 0.5 and powder/sand-ratio = 1:3 for all mixes.

Three binders are investigated:
1) Reference (co-ground clinker and gypsum, i.e. only cement as binder),
2) ALS-substituted, co-ground (co-ground clinker, gypsum and ALS),
3) ALS-substituted, blended (co-ground clinker and gypsum, subsequently blended with ALS).

The level of substitution was 10% ALS relative to the total binder mass (cement + ALS).

Table 4. Mix proportions of mortars (mass [g]).

	Clinker (95%) + gypsum (5%)	ALS	CEN- standard Sand	Demineralized Water
Reference	450	0	1350	225
ALS-substituted	405	45	1350	225

3.2 Processing of the raw materials

3.2.1 Ball mill parameters

Ball mill jar dimensions are listed in Table 5. The ball mill jar is filled with 50 vol-% balls and 25 vol-% raw materials. It is possible to operate three ball mills simultaneously. In order to process all the received clinker, the batches shown in Table 6 were made.

Table 5. Ball mill jar dimensions.

Diameter	21 cm
Length	16 cm
Volume	5.5 ℓ

Table 6. Grinding Batches for each binder.

	Volume of grinding material (ℓ)		Grinding Batches
	Cement	ALS	
1. Reference	8.79	0	6
2. ALS-substituted, co-ground	7.95	3.24	8
3. ALS-substituted, blended	7.95	0	6

Table 7 shows the approached, optimal diameter distribution of the balls.

Table 7. Diameter distribution of the balls.

	Diameter	Volume
Large Balls	35 mm	45%
Middle Balls	25 mm	10%
Small Balls	15 mm	45%

The ball milling was done at 50-75% of the critical speed which is the outer layer of the media centrifuging against the wall.

$$\text{Critical speed} = \frac{42.4}{\sqrt{D}}\,\text{rpm}$$

D is the diameter of the ball mill (0.21m). Critical speed according to this equation is 93 rpm. 65% of this value is 60 rpm i.e. 1 revolution per second.

3.2.2 Procedure of ball milling

The clinker and gypsum are initially crushed with a jaw crusher and subsequently mixed homogeneously in the mass proportions 95 to 5, see Fig. 1. ALS is dried to constant mass at 105 ^{o}C before use. Grinding is done by ball milling. For every 1 hour ball milling of binder 1 (reference) the Blaine specific surface and the sieve residue at the 50 µm sieve was measured. Ball milling was continued until a Blaine specific surface in the range 300-400 m^2/kg was reached. For binder 2 (co-ground ALS) the same ball milling time as for binder 1 is used (i.e. same energy input) since the Blaine specific surface is not a reliable measure for the cement particle size in a composite powder. For binder 3 (blended ALS), ALS is blended together with the cement in the mortar mixer.

Fig. 1. The clinker and gypsum are initially crushed with a jaw crusher (left) and subsequently mixed homogeneously (right).

3.3 Blaine measurement

The test is done by a HMK-C1 Cement Blaine Fineness Air Permeability Apparatus according to standard DS/EN 196-6, see Fig. 2. As a reference material ordinary white Portland cement from Aalborg Portland with a specific surface area of 400 m^2/kg was selected. The time that it takes the liquid in the manometer to descend a fixed distance is measured for both the reference material and the actual material, and from this the specific surface area of the actual material can be calculated as shown in eq. (1):

$$S = \frac{S_S \sqrt{T}}{\sqrt{T_S}} \tag{1}$$

where
S is the specific surface area [m^2/kg] of the material under test,
S_s is the specific surface area [m^2/kg] of the reference material,
T is the time [s] of air flow of the material under test, and
T_S is the time [s] of air flow using the reference material.

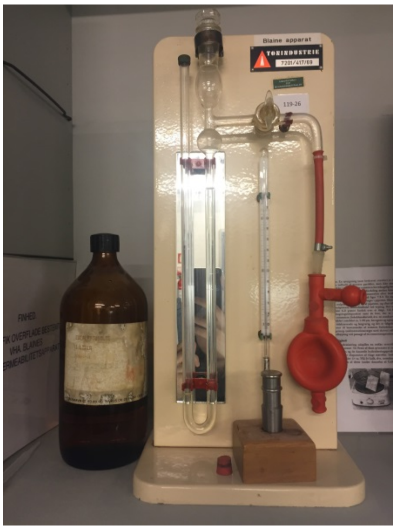


Fig. 2. HMK-C1 Cement Blaine Fineness Air Permeability Apparatus.

The tesing temperature is 21.7°C and the relative humidity is 65%. One set of permeability test was done twice for both two beds (four measurements for one set). For both of the two samples the means of the times and temperatures are calculated. After ball milling binder 1 (reference) for two hours, the specific surface area, S, was 278 m^2/kg. After a further two hours of ball milling, S was 358 m^2/kg which is in the desired range 300-400 m^2/kg. Therefore, this total ball milling time (4 hours) is used for the rest of the batches. The specific surface and 50 μm sieve residue of reference and co-ground ALS are listed in Table 8.

Table 8. The specific surface and 50 μm sieve residue of reference and co-ground ALS

	Reference						co-ground ALS
Grinding batch	1	2	3	4	5	6	1
Specific surface (m^2/kg)	358	380	367	365	360	370	387
50 μm sieve residue (%)	0.39	0.35	0.37	-	-	-	0.01

3.4 Mortar mixing

Mixing was done in a 20 ℓ epicyclic mixer, see Fig. 3. For ALS-substituted, blended, cement and ALS were initially dry mixed in the mixer for 5 min. For the other two mixes no initial mixing of the binder was relevant. A low mixer speed (140 ± 5 min^{-1}) was used during addition of the water to the binders. After 30 s of paste mixing, the sand was added during the subsequent 30 s. The mixer was switched to high speed (285 ± 10 min^{-1}) and mixing was continued for another 30 s. The mixer was stopped for 90 s to remove the mortar adhering to the walls of the bowl and mixing was subsequently continued at the high speed for a further 60 s.

Immediately after mixing the flow table test was done, followed by measurement of the fresh air content by the air pressure method.

Fig. 3. Mixing process: 20 ℓ epicyclic mixer (left) and freshly mixed mortar (right).

3.5 Flow test

Flow is measured according to ASTM C1437-13. Two layers of fresh mortar were filled into the conical standard mould and each layer tamped 20 times. The excess mortar was wiped off with a metal rod by a sawing motion. Then the mould was removed and the table was immediately dropped 25 times in 15 s. The flow was measured by two orthogonal diameters of the mortar, see Fig. 4.

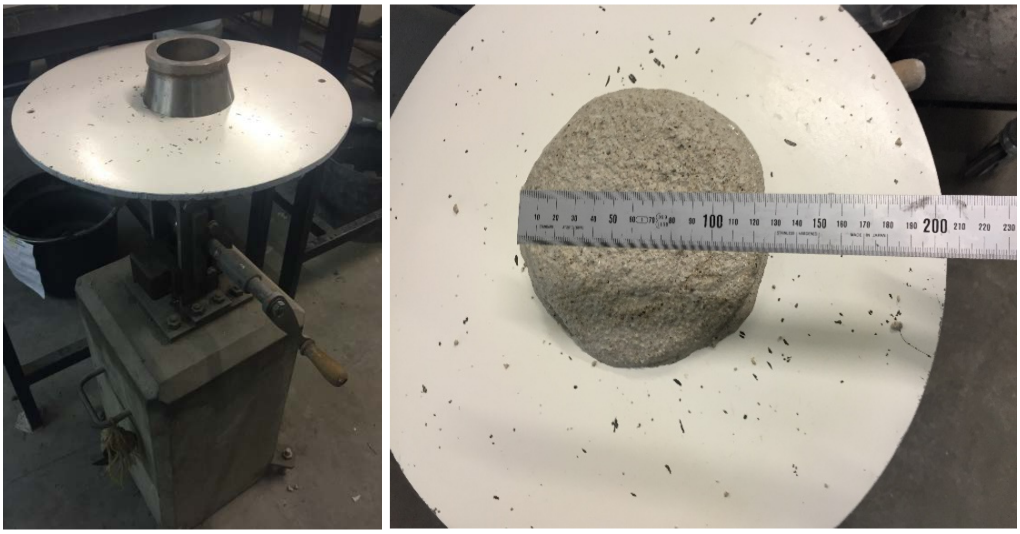


Fig. 4. Flow table (left) and flow measurement represented by the diameter of the fresh mortar (right).

3.6 Air content measurement

3.6.1 Air content by pressure meter measurement

Air content is measured on the fresh mortar by the pressure method, ASTM C231/C231M-14. After the flow table method was completed, mortar was filled into the 1 ℓ pressure meter seen in Fig. 5. The filling was done in three equal layers, tamped 25 times each and the pressure meter was placed on a vibrating table (EN 196-1 Vibrating table B, nominal 50 Hz) for a total of 120 s in order to remove encapsulated air. The excess mortar was removed by a metal rod with a sawing motion. The edge of the pressure meter bucket was cleaned and the lid was closed. Water was injected into the left valve until it flowed out of the right valve. The valves were closed at once. The meter was pressurized to the required level and released to expose the mortar and thereby measure its air content in the fresh state.

Fig. 5. Pressure meter for measuring the air content in fresh mortar.

3.7 Mortar casting, demoulding and curing

Casting was done in standard mortar molds 4×4×16 cm^3. The molds were placed on a vibrating table and the fresh mortar was filled in two layers and vibrated for a total of 120 s to remove encapsulated air. A plastic cover was placed on the molds and each covered mold was transferred to a room with 95% RH, 21°C. The mortar specimens were demolded 1 day after casting. After demolding each mortar specimen was weighed over and under water and immediately thereafter immersed in lime saturated water until further testing, see Fig. 6. Curing temperature of the lime water was 19°C.

Fig. 6. Fresh mortar casted in moulds (left) and mortar samples immersed in lime water (right).

3.8 Air content by point counting method

This is a statistical method for determination of air content on hardened mortar and concrete according to ASTM C457. The test specimen (4×4×16 cm^3) was cut along the short edge into four pieces and the surface to be examined was subsequently finely ground at grit #220 SiC paper. A stereomicroscope (Nikon SMZ-2T) at magnifications from 1× to 6.3× equipped with a Pelcon automatic point counter was used to analyze air content of the test specimen, as shown in Fig. 7.

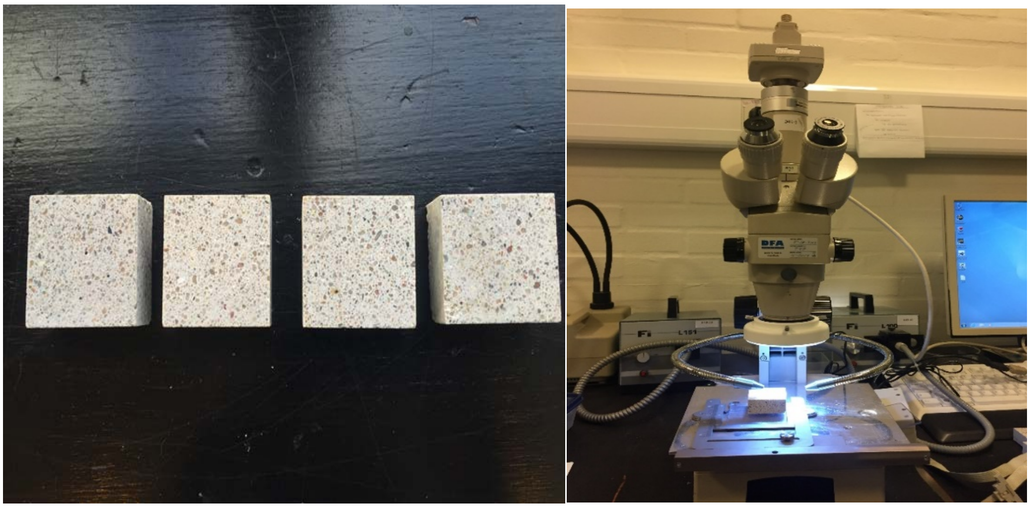


Fig. 7. Test specimen cut into 4 pieces (left) and entire apparatus (right) for point counting by stereomicroscopy.

The area of analysis for each piece of sample was 30 mm × 30 mm. During the analysis, the motorized stage moves the sample by one step-length (set to 2.00 mm) when pressing one of the key-pad buttons. By the end of each test-line, the sample is moved manually to the next test-line by means of two toothed sliding wheels. For each piece of test sample, 225 points were counted (15 lines with 15 points). On one test specimen in total 900 points were thus being counted. By selecting an index point in the reticule of the eyepieces of the stereomicroscope, 1 or 2 were typed in on a key-pad depending on whether the reference point did or did not fall within an air void. Finally, by summing up the total number of air voids observed in the four pieces, the air content was calculated as the percentage of the number of air voids out of 900 points. The number of air void counts for each piece of mortar are listed in Table 9.

Table 9. The times of observing air for each piece of mortars.

	1st	2nd	3rd	4th	Air content (%)
Reference	6	8	4	7	2.8
ALS-substituted, co-ground	8	11	12	11	4.67
ALS-substituted, blended	8	4	5	8	2.78

3.9 Mechanical testing

The mechanical testing carried out generally followed EU standard EN 196-1. In accordance with the EU standard, flexural strength measurements were done at a minimum of 3 samples at each test time. The flexural strength of the hardened mortar was determined by three-point loading of a prismatic specimen. Subsequent to the flexural failure of the specimen the compressive strength was determined on each half of the prism. The rate of load for flexural strength and compressive strength were 50 ± 10 N/s and 2400 ± 200 N/s respectively. The mechanical measurements were made at 1, 2, 3, 7, 14, 28, 56 and 112 days.

Fig. 8. Flexural strength measured by three-point loading of a prismatic specimen (left) and subsequent compressive strength measurement on one of the half prisms (right).

3.10 Scanning electron microscopy and Energy Dispersive X-ray Analysis

Scanning electron microscopy and energy dispersive X-ray analysis (element mapping) were performed on some of the powder materials in order to examine to what extent the ALS agglomerates were broken by the co-grinding process.

4. Results and analysis

Table 10 lists the results of consistency of fresh mortar by flow table together with air content of fresh and hardened mortars. It should be noted that the result of air content determination by weighing is an average value of all tested specimens; raw data are listed in appendix II.

Table 10. Test results for fresh and hardened mortars. *Two flow test measurements perpendicular to each other were made on each mixture.

	Reference	ALS-substituted, co-ground	ALS-substituted, blended
Flow test (mm)*	225, 223	205, 201	160, 162
Flow percentage (%)	124	103	61
Air content by pressure meter (%)	3.6	4.1	4.7
Air content by weighing (%)	0.86	2.20	4.87
Air content by point counting (%)	2.78	4.67	2.78
Density (g/cm^3)	2.33	2.26	2.19

To estimate the air content of the mortars two methods – weighing and the pressure method – were initially used, since this parameter is important for the analysis of the measured mechanical properties. The results from these two methods are shown in Fig. 7. There seems to be a linear relation between these, however, the results do not follow the 1:1 relation for the reference and the ALS-co-ground mortar. In order to supplement the data of these two test methods, a third technique was adopted, the point counting method, for the hardened reference mortar. As seen from Table 10 there is not a good agreement between the different techniques for determination of air content. Uncertainties are related to all techniques, see e.g. [2]. Air content determined by weighing is selected for further analysis of strength results, since the weighing method is done on hardened samples (which is relevant for strength analysis) and since the these results follow the same trend as for the rheology data.

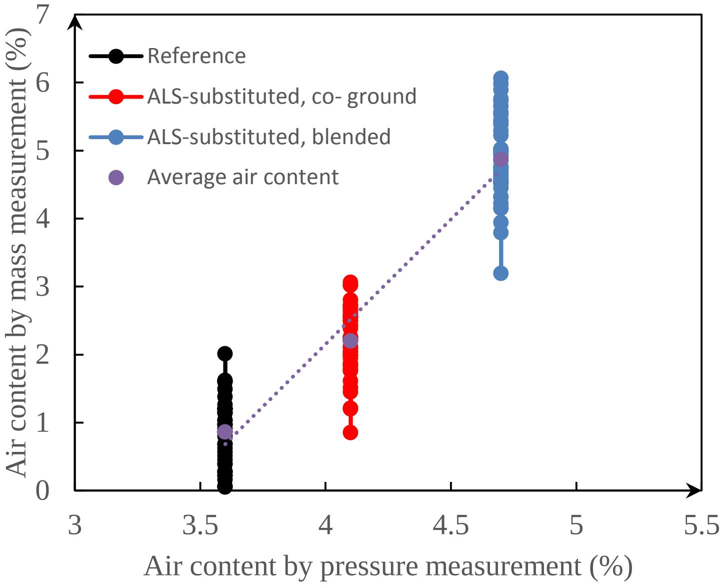


Fig. 7. Air content of fresh mortars by pressure measurement vs.
hardened mortars by weighing.

The relationship between flow value and air content by the weighing method of the three mortars are given in Fig. 8. Clearly ALS substitution of cement leads to a lower flow percentage and a higher air content, most likely due to the stiffer mortar which more easily encapsulates air during mixing and which is less likely to release it again during casting and vibration.

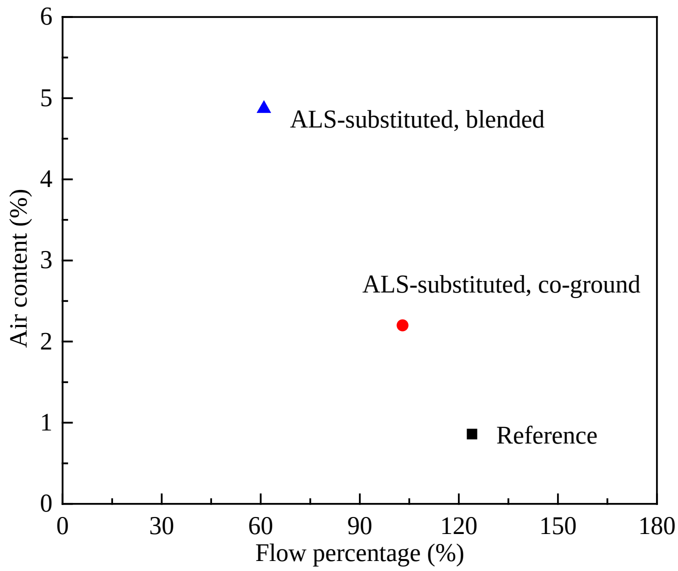


Fig. 8. Flow percentage of fresh mortars vs. air content by weighing.

The flexural strength of mortars (reference, ALS-substituted, co-ground and blended) is shown in

Table 11 and Fig. 9. Taking into account the fluctuations on the individual measurements and the measurements as a time series, there does not seem to be a marked difference in the flexural strength between the three mixes. There may be a slight tendency towards a lower flexural strength of ALS-substituted, blended, and perhaps a slight tendency towards a higher flexural strength of ALS-substituted, co-ground, but the effect, if any, is minor.

Table 11. Flexural strength [MPa] of the three mortars.

Time (d)	Reference	ALS-substituted, co-ground	ALS-substituted, blended
1	2.9 ± 0.22	3.5 ± 0.14	3.4 ± 0.24
2	5.0 ± 0.35	5.2 ± 0.28	5.0 ± 0.25
3	6.7 ± 0.21	6.9 ± 0.11	6.7 ± 0.16
7	7.6 ± 0.24	8.2 ± 0.20	7.7 ± 0.35
14	9.0 ± 0.57	9.2 ± 0.37	8.7 ± 0.43
28	10.5 ± 0.37	9.7 ± 0.52	9.1 ± 0.42
56	10.7± 0.18	10.7 ± 0.27	9.9 ± 0.05
112	11.2 ± 0.60	11.5 ± 0.32	10.6 ± 0.42

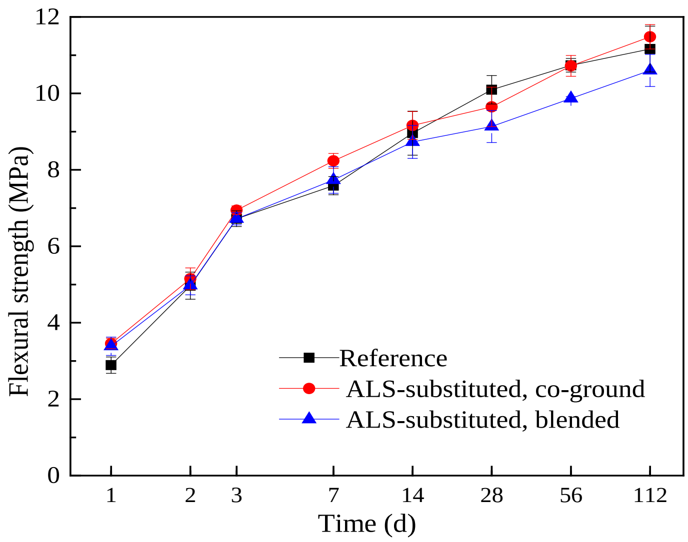


Fig. 9. Flexural strength of the three mortars.

Table 12. Compressive strength [MPa] of three mortars.

Time (d)	Reference	ALS-substituted, co-ground	ALS- substituted, blended
1	17.7 ± 1.3	18.3 ± 0.01	17.1 ± 0.8
2	31.9 ± 1.1	31.5 ± 1.3	27.7 ± 0.9
3	43.1 ± 1.4	40.6 ± 0.01	36.6 ± 2.0
7	56.3 ± 2.0	54.1 ± 1.6	50.4 ± 0.7
14	69.0 ± 2.3	59.8 ± 1.8	56.4 ± 2.5
28	76.5 ± 1.9	68.0 ± 1.5	66.6 ± 2.3
56	83.8 ± 3.0	77.7 ± 3.1	74.6 ± 0.9
112	96.5 ± 3.2	91.7 ± 3.7	83.5 ± 2.9

Table 13. Compressive strength [MPa] of three mortars corrected by weighing.

Time(d)	Reference	ALS-substituted, co-ground	ALS- substituted, blended
1	18.5 ± 1.3	20.52 ± 0.01	22.54 ± 0.8
2	33.3 ± 1.1	35.38 ± 1.3	36.56 ± 0.9
3	45.0 ± 1.4	45.56 ± 0.01	48.33 ± 2.0
7	58.8 ± 2.0	60.78 ± 1.6	66.57 ± 0.7
14	72.1 ± 2.3	67.15 ± 1.8	74.61 ± 2.5
28	79.9 ± 1.9	76.39 ± 1.5	88.05 ± 2.3
56	87.5 ± 3.0	87.29 ± 3.1	98.59 ± 0.9
112	100.9 ± 3.2	103.07 ± 3.7	110.38 ± 2.9

The compressive strength of mortars (reference, ALS-substituted, co-ground and blended) is shown in Fig.10. It can be seen that the samples ALS-substituted, co-ground and blended have a lower compressive strength throughout the considered time range. Compared with the reference mortar the reduction in compressive strength of ALS-substituted, co-ground and blended at 28 days were 11% and 15% respectively. The strength reduction for ALS-substituted, co-ground is less pronounced than for ALS-substituted, blended. This may have several explanations including a better dispersion of co-ground ALS and thus better reactivity of the ALS and better pore filling in between the cement particles. Also breakage of ALS agglomerates and dispersion of the particles may enable them to better act as nucleating sites and thus improve the hydration progress of the cement. Another important factor concerns the observed, different air contents which is related to the differences in workability, and apparently also strongly influenced by the presence and form of ALS present in the mortar. It is known that an increase in air content of 1% leads to a reduction in compressive strength by 5%. Therefore, by taking the factor of air content into consideration, the results of compressive strength can be corrected for differences in air content. Such corrected results are shown in Fig. 11. This comparison is relevant as it will be possible to correct the workability of the different mortars by means of plasticizer adjustment, and thus most likely eliminate the differences in air content.

For the corrected results in Fig. 11 it is seen that the strength reduction by ALS substitution apparently can be fully accounted for by the differences in air content of the samples.

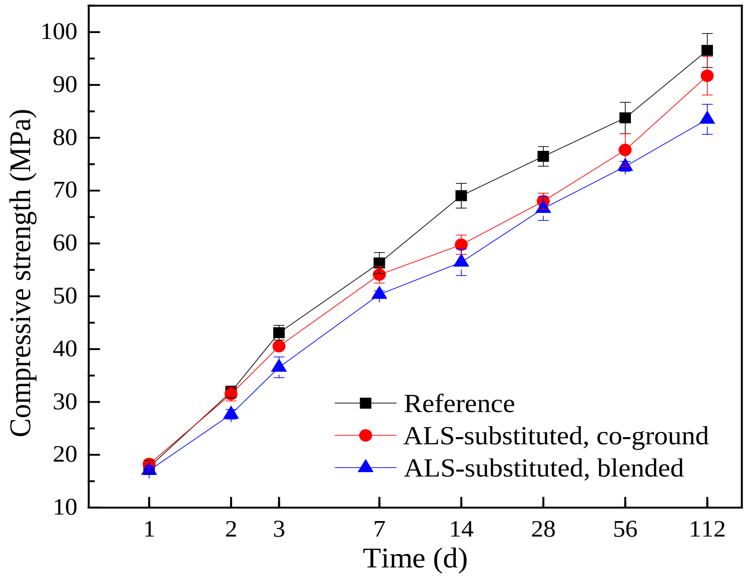


Fig. 10. The effect of ALS substitution, co-grounded and blended on the compressive strength of hardened mortars.

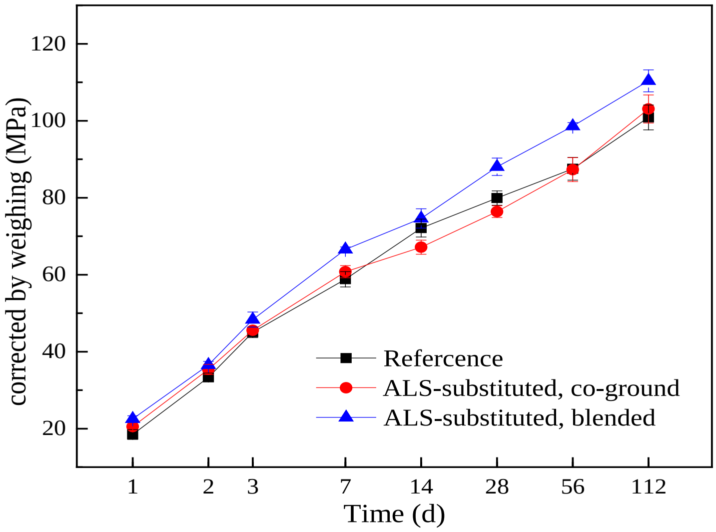


Fig. 11. The effect of ALS substitution, co-grounded and blended on the compressive strength of hardened mortars. The results are corrected for the influence for air measured by weighing.

SEM images were captured to investigate if the ALS agglomerates were broken by the co-grinding process, see Figs. 12 and 13. As seen from Fig. 12 (top left), and in accordance with the data sheet Appendix 1, the ALS powder consist of agglomerates of particles mainly in the size range from a few µm to about 50 µm. Relative to the ALS powder, the cement particles are slightly finer, perhaps the typical particle size of the cement is 4 times smaller. From the EDAX maps it is possible to identify main components in the ALS co-ground system: C_3S (high Ca, some Si), C_2S (medium high Ca, some Si), C_3Al (high Ca, some Al), ALS (no Ca, high Si), gypsum (high S). By further comparison with the SEM image in Fig. 13 and SEM images in Fig. 12 it seems clear that in the ALS co-ground binder the ALS agglomerates have been broken down to particle sizes of a few µm. The ALS particles appear to be well dispersed in the cement system. These microscopical observations are in accordance with the registered differences regarding flowability and air contents – and further the mechanical properties – between ALS-substituted, co-ground and blended.

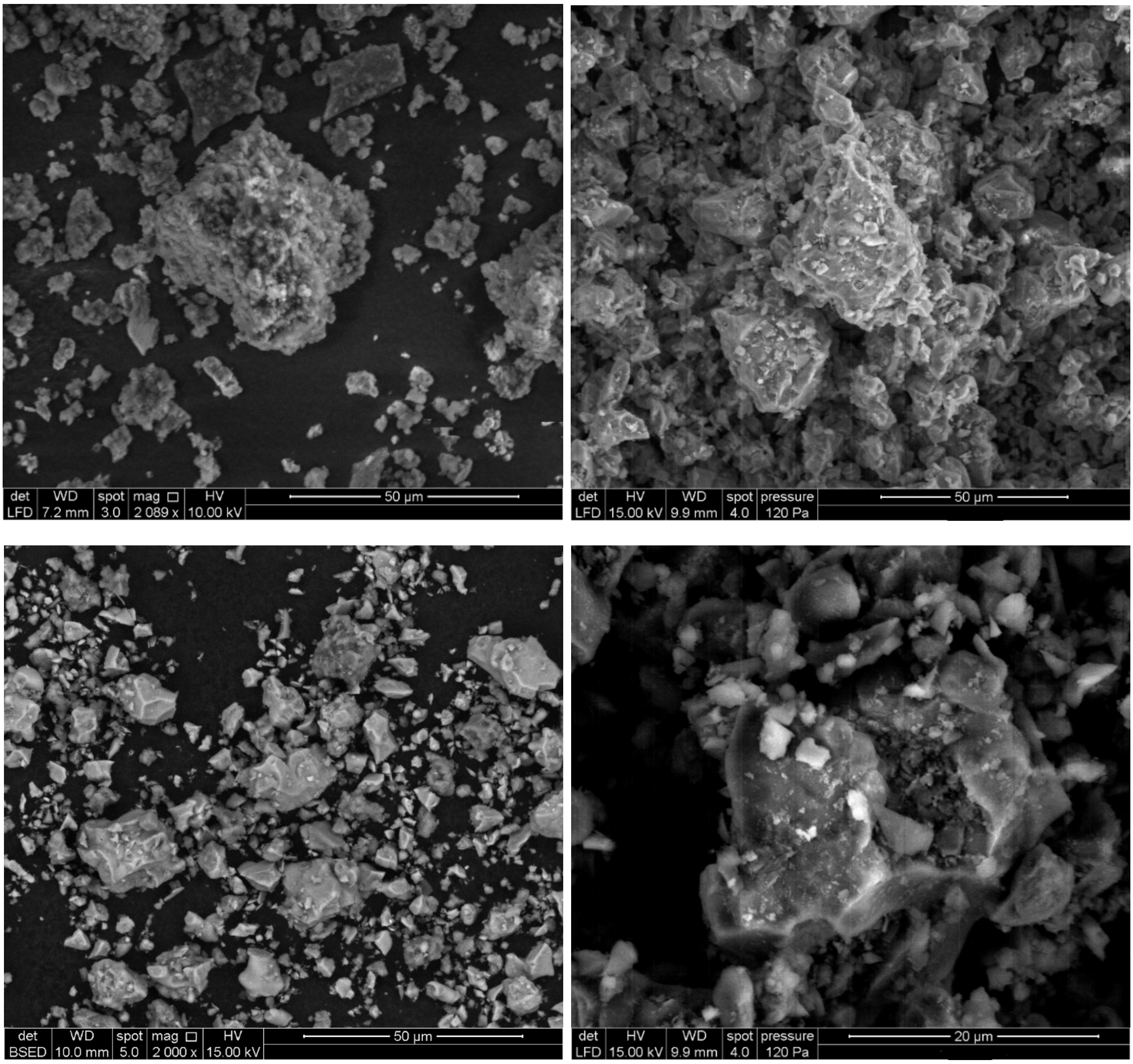
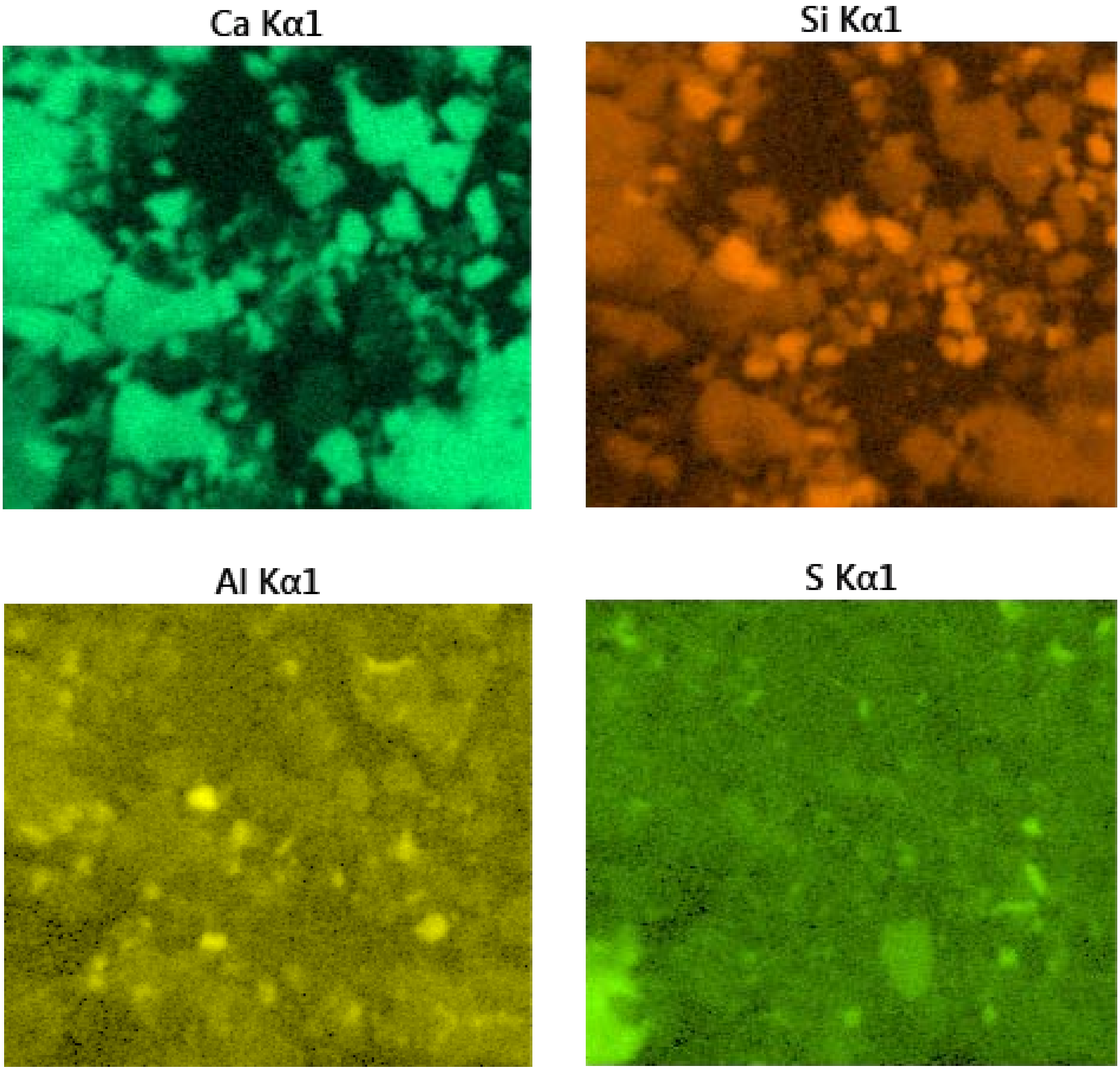


Fig.13. SEM images of the different powders. Top left: ALS as received (i.e. after fluoride stabilization). Top right: Cement, i.e. ground cement clinker with gypsum. Bottom left and right: Cement with co-ground ALS at two different magnifications (cf. scale bar).



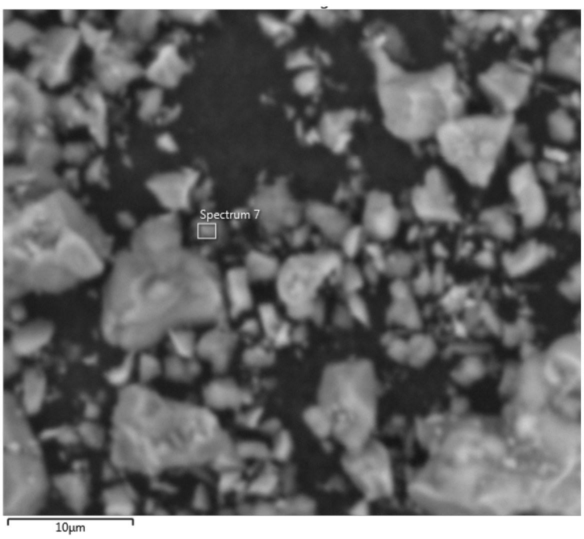


Fig. 14. Identical view fields by SEM (bottom left) and four EDAX mappings of the produced ALS-substituted powder. The elements analyzed for are shown on the EDAX mappings. Image height is approximately 40 µm.

5. Conclusion

The following conclusions can be drawn based on the present laboratory investigation.

1. The fluoride in the ALS seems within the tested regime to be effectively bound as CaF_2 and does apparently not disturb the cementitious reactions.
2. Co-grinding of ALS and cement clinker (and gypsum) leads to a finer dispersion of ALS in the cementitious system and breaks up ALS agglomerates from about 5-50 µm size to about 1-5 µm size.
3. The inclusion of ALS in the mortar as a mineral admixture with the substitution ratio of 10% of cement mass resulted in a higher air content and a corresponding reduction in the flow compared with the reference mortar. Compared with blending ALS during the mixing, mortar containing co-ground ALS was closer to the reference mortar considering these properties.
4. The addition of ALS did not affect the flexural strength significantly at the investigated hardening times 1 to 122 days.
5. Mortars containing 10% ALS by cement mass exhibited a lower compressive strength compared with the reference mortar. At 28 days the strength reduction is 11% for ALS-substituted, co-ground and 15% for ALS-substituted, blended. Apparently the strength reduction can be accounted for by the increased air content, i.e. it is expected that it by simple means will be possible to incorporate ALS in cementitious systems without strength loss.

References

DS/EN 196-3+ A1 Methods of test of testing cement- Part 3: Determination of setting time and soundness

DS/EN 196-6 Method of testing cement- Part 6: Determination of fineness

EN 196-1 Methods of test of testing cement- Part 1: Determination of strength

DS/EN 480-1 Admixture for concrete, mortar and grout- Test methods- Part 1: Reference concrete and reference mortar for testing

DS/EN 1015-10 Methods of test for mortar for masonry- Part 10: Determination of dry bulk density of hardened mortar

Standard Test Method for Air Content of Freshly Mixed Concrete by the pressure (ASTM C231/C231M-14)

Standard Test Method for Flow of Hydraulic Cement Mortar (C1437-13)

Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete (ASTM C457/C457M − 12)

[1] R.S.Bundgaard and S.S.Kjær, Alusilica as a Mineral Admixture in Concrete, BSc thesis, Technical University of Denmark, Department of Civil Engineering, 2015.

[2] P.Lawrence, E.Ringot and B.Husson, About the measurement of the air content in mortar, Materials and Structures, 32, October 1999, pp. 618-621.

Appendix I Specification of Alusilica

Specification – Alusilica

Our aluminium fluoride production process generates silicon dioxide (SiO_2). Main application today is as fluorine absorbent in the phosphoric acid industry for the production of fluorosilicic acid. Silicon dioxide is also used in other applications in the chemical and mineral industry – such as for improving properties in cement.

Typical Analysis (dry sample)	SiO_2	85 %
	Al	2 %
	F	5 %
	LOI	Rest
	By delivery the products contains 25–30 % moisture	
Bulk density (typical)	By filling	300 kg/m3
	By shaking	500 kg/m3
Sieve analysis (typical)	>50µm	5 %
	5–50 µm	90 %
	< 5µm	5 %
Other properties	Alusilica is a synthetic, amorphous and dustless silicon dioxide.	
Colour:	white	
pH:	2.5–4 (25 % water mix)	
Angle of repose by filling:	40°	

alufluor

HIGHER EFFICIENCY, LOWER IMPACT SINCE 1973

P.O. Box 902 SE-251 09 Helsingborg, Sweden

Tel +46 42 17 10 20 Fax +46 42 13 52 85 www.alufluor.com alufluor@alufluor.com

Appendix II

Air content (%) calculated based on measurements of mass, m, and weight when suspended in water, w, on hardened mortar samples.

	Reference			ALS-substituted, co-ground			ALS-substituted, blended		
	m (g)	w (g)	Air content (%)	m (g)	w (g)	Air content (%)	m (g)	w (g)	Air content (%)
1	578	330	0.97	570	316	2.68	570	309	5.29
2	580	331	0.91	571	317	2.5	568	310	4.52
3	590	337	1.03	575	320	2.21	570	311	4.55
4	578	330	0.97	577	322	1.87	578	316	4.32
5	577	330	1.2	572	317	2.72	564	305	5.56
6	577	328	0.39	568	314	3.02	568	311	4.15
7	578	329	0.57	574	319	2.38	568	306	5.98
8	579	328	0.06	577	321	2.25	565	307	5.02
9	575	327	0.45	596	332	2.09	566	307	5.22
10	591	339	1.6	568	314	3.02	572	311	4.95
11	586	335	1.14	575	320	2.21	567	309	4.69
12	577	328	0.39	584	327	1.45	574	312	4.98
13	576	327	0.22	579	321	2.67	570	311	4.55
14	581	330	0.28	576	320	2.42	583	318	4.59
15	581	331	0.68	571	318	2.12	578	312	5.76
16	578	328	0.16	573	318	2.55	574	312	4.98
17	582	332	0.86	568	316	2.25	580	314	5.44
18	574	326	0.27	567	314	2.8	580	316	4.72
19	579	331	1.15	572	319	1.95	576	313	5.02
20	578	331	1.38	575	320	2.21	574	312	4.98
21	576	328	0.62	595	333	1.51	576	312	5.38
22	577	330	1.2	598	334	1.76	577	314	4.85

23	583	333	1.03	570	315	3.06	576	311	5.73
24	584	334	1.2	585	325	2.42	576	314	4.65
25	584	333	0.8	572	317	2.72	570	308	5.65
26	580	330	0.51	573	318	2.55	574	309	6.06
27	574	326	0.27	573	320	1.78	567	307	5.42
28	585	331	0.22	583	328	0.85	579	318	3.79
29	586	332	0.05	608	340	1.61	560	305	4.76
30	582	333	1.26	602	336	1.85	562	303	5.89
31	577	331	1.62	606	340	1.2	562	304	5.53
32	584	334	1.2	580	322	2.5	570	310	4.92
33	591	340	2.01	574	320	1.99	571	311	4.75
34	588	337	1.49	577	321	2.25	575	314	4.45
35	579	331	1.15	576	321	2.04	568	311	4.15
36	591	339	1.6	574	320	1.99	572	313	4.22
37	585	334	0.97	575	319	2.59	567	313	3.19
38	582	332	0.86	575	321	1.82	574	313	4.62
39	582	330	0.06	574	322	1.21	568	310	4.52
40	574	327	0.68	576	321	2.04	579	316	4.52
41	584	335	1.61	577	320	2.63	567	310	4.32
42	584	334	1.2	573	318	2.55	567	311	3.94